POLYURETHANE FOAM FROM OIL PALM FRUIT WASTE: SYNTHESIS AND CHARACTERIZATION OF BIOPOLYOL AND FOAM PROPERTIES

SHAHARUDDIN BIN KORMIN

A thesis submitted in
fulfilment of the requirement for the award of the
Doctor of Philosophy

Faculty of Mechanical and Manufacturing Engineering
Universiti Tun Hussein Onn Malaysia

AUGUST 2018

In the name of ALLAH, Most Gacious, Most merciful. Muhammad S.A.W

This thesis especially dedicated to:

My beloved late father
#Tuan Hj Kormin Bin Mat Ngali

My beloved mother
Puan Hjh Mariam Binti Parno

My beautiful wife
Fatin Amira Binti Mohd Juhan

My cute son
Muhammad Uwais Al Qarni

My supportive supervisor
Assoc. Prof. Dr. Anika Zafiah Binti Mohd

Rus Thank you for everything

ACKNOWLEDGEMENT

In the name of Allah, the Most Gracious and Most Merciful

Alhamdulillah, all praises to Allah for the strengths and His blessing in completing this thesis. I would like to express my deepest gratitude and appreciation to my supervisor Assoc. Prof. Dr. Anika Zafiah Mohd Rus for her continuous guidance, valuable advice and freely given her time to share her expert knowledge throughout my research project.

I would also like to extend my sincere thanks to all lecturer, lab assistants namely to En Fazlannuddin Hanur bin Harith, En Mohd Tarmizi bin Nasir, En Anuar bin Ismail and my colleagues for their help and assistance. The assistance and technical support at Faculty of Mechanical and Manufacturing Engineering (FKMP); Materials Science Laboratory and Polymer Ceramics Laboratory, Faculty of Civil and Environmental Engineering (FKAAS) and Faculty of Applied Science and Technology (FAST) are greatly appreciated. My thanks to all the assistance and services provided by Centre of Graduate Studies, computer facilities in Universiti Tun Hussein Malaysia (UTHM) and UTHM Library are acknowledged. Also, I want to say my special thanks to my late father, mother, wife, son, brother and sister for their endless love, prayers and encouragement. Thank you very much.

Last but not least, thank you to everyone that had involved directly and indirectly for all inspirations and directions.

Thank You.

ABSTRACT

Oil palm industries generate abundant amount of biomass, which when properly used will not only be able to solve the disposal problem but also can create value added products. The aim of this research is to produce of polyurethane (PU) foams with biopolyols from liquefied oil palm fruit waste (PW). Three parts of oil palm fruit waste (PW): oil palm mesocarp fiber (PM), oil palm shell (PS) and oil palm kernel (PK) was liquefied using liquefaction solvent with sulfuric acid as catalyst was studied. PU foams were prepared from 100 % of PM biopolyol (PMF), incorporation of liquefied PM biopolyol with renewable monomer (PMRF), epoxy (PMEF) and PM fiber filler (PMF_{1-9}) in the presence of dibutyltine dilaurate as a catalyst with water as blowing agent, and silicon oil as a surfactant. The liquefied product and polyurethane foam samples were characterized through the physical, chemical, thermal, mechanical and morphological analysis. The optimal liquefaction conditions were determined to be PW/PEG400 = 1/3, 5 % acid loading, and liquefaction at 150 °C for 120 min. The results revealed that more than 50 % of the oil palm fruit waste converted into liquefied product. The GC-MS analysis showed that the chemical components of phenol and its derivatives, organic acids, hydrocarbon, ester, benzene groups and alcohols. FTIR spectroscopy analysis demonstrated the formation of urethane linkage in liquefied PW biopolyol which suitable for the production of PU foams. Meanwhile, In terms of the thermal properties, the improved thermal insulation properties were achieved at a composition of PMF foams. PMF foams showed higher compression strength (61.41 kPa) and tensile strength (117 kPa). In DMA result, the higher crosslinking density (33.17 M/m^3) and crosslinking interaction (NHC(O)O) in PU foam was determined. SEM revealed the exfoliated structure of PU foams and indicated the cells within the obtained foams are closed cells. The properties of the PU foam were indicating that the liquefied PM biopolyol from a solvolysis liquefaction could be successfully applied to fabricate PU foam products as a substitute for industrial foams with lower cost.

ABSTRAK

Industri kelapa sawit menjana banyak biomas yang apabila digunakan dengan baik, bukan sahaja dapat menyelesaikan masalah pelupusan tetapi juga dapat menghasilkan produk nilai tambah. Tujuan penyelidikan ini adalah untuk menghasilkan busa poliuretana (PU) dengan biopoliol dari sisa buangan cecair kelapa sawit (PW). Tiga bahagian sisa buangan kelapa sawit (PW): gentian mesokarp kelapa sawit (PM), tempurung kelapa sawit (PS) dan kernel kelapa sawit (PK) dicairkan menggunakan pelarut pencairan dengan asid sulfurik sebagai pemangkin telah dikaji. Buih buih disediakan dari 100 % PM biopoliol (PMF), penggabungan cecair PM biopoliol dengan monomer yang diperbaharui (PMRF), epoksi (PMEF) dan pengisi gentian PM ($PMF_{1\text{-}9}$) dengan kehadiran dibutyltine sebagai pemangkin dengan air sebagai agen pengembang, dan minyak silikon sebagai surfaktan. Sampel produk cecair dan poliuretana telah dicirikan menggunakan analisis fizikal, kimia, haba, mekanikal dan morfologi. Keadaan pencecairan optima telah ditentukan oleh sampel dengan PW/PEG400 = 1/3, 5 % muatan asid, dan pencecairan pada 150 °C selama 120 minit. Hasilnya menunjukkan bahawa lebih daripada 50 % sisa buah kelapa sawit ditukar kepada produk cecair. Analisis GC-MS menunjukkan bahawa komponen kimia fenol dan terbitannya, asid organik, hidrokarbon, ester, kumpulan benzena dan alkohol. Analisis spektroskopi FTIR menunjukkan pembentukan hubungan uretana dalam cecair PW biopoliol yang sesuai untuk menghasilkan busa PU. Sementara itu, dari segi sifat terma, sifat penebat terma yang lebih baik telah dicapai pada komposisi busa PMF. Busa PMF menunjukkan kekuatan mampatan (61.41 kPa) dan tegangan (117 kPa) yang tinggi. Dalam keputusan DMA, hubungan silang yang lebih tinggi (33.17 M/m^3) dan interaksi silang (NHC(O)O) dalam busa PU telah ditentukan. SEM menunjukkan struktur busa yang terkelupas dan menunjukkan sel-sel dalam busa yang diperolehi adalah sel tertutup. Sifat busa PU menunjukkan bahawa cecair PM biopoliol dari pencecairan solvolisis boleh digunakan dengan berkesan untuk menghasilkan produk busa PU sebagai pengganti busa industri dengan kos yang lebih rendah.

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LIST OF SYMBOLS AND ABBREVIATIONS

cm	-	Centimetre
cm^{-1}	-	Per centimetre
^{o}	-	Degree
^{o}C	-	Degree Celsius
$^{o}C/min$	-	Degree Celsius per minute
E'	-	Storage modulus
E''	-	Loss modulus
g	-	Gram
g/mol	-	Gram per mol
h	-	Hour
CrI	-	Crystallization Index
kPa	-	Kilo Pascal
MPa	-	Mega Pascal
Mw	-	Weight average Molecular weight
Mn	-	Number average Molecular weight
Nv	-	Pore density per volume
mL	-	Millilitre
mm	-	Millimetre
J	-	Joule
n	-	Number of pore
rpm	-	Revolutions per minute
K	-	Temperature in Kelvin
T	-	Absolute temperature
T_g	-	Glass transition
V_e	-	Cross-linked density
W_W	-	Mass of an immersed and suspended specimen in air
W_D	-	Mass of air dried specimen

W_S	-	Mass of immerse and suspend specimen in liquid
%	-	Percentage
μm	-	Micrometre
nm	-	Nanometre
θ	-	Theta
δ	-	Delta
λ	-	Wavelength
ρ	-	Density
ASTM	-	American Society for Testing Materials
ATR	-	Attenuated total reflection
DTG	-	Differential Thermogravimetric Analysis
DTA	-	Differential Thermal Analysis
DMA	-	Dynamic Mechanical Analysis
DSC	-	Differential Scanning Calorimetry
E	-	Epoxy
EF	-	Epoxy Foam
EG	-	Ethylene glycol
EDX	-	Energy Dispersive X-ray Spectroscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
GCMS	-	Gas Chromatography-Mass Spectrometry
GPC	-	Gel Permeation Chromatography
Gly	-	Glycerol
H_2O	-	Water
H_2SO_4	-	Sulphuric acid
MDI	-	Methylene Diphenyl Diisocyanate
NaOH	-	Natrium hydroxide
OM	-	Optical Microscope
PA	-	Polyhydric Alchohol
PM	-	Oil Palm Mesocarp Fiber
PS	-	Oil Palm Shell
PK	-	Oil Palm Kernel
PU	-	Polyurethane
PDI	-	Polydispersity Index
PEG	-	Polyethylene glycol

PW	-	Oil Palm Fruit Waste
PMF	-	Oil Palm Mesocarp Fiber Foam
PMRF	-	Oil Palm Mesocarp Fiber/Renewable Polymer Foam
PMEF	-	Oil Palm Mesocarp Fiber/Epoxy Foam
R	-	Renewable Monomer
RF	-	Renewable Polymer Foam
SEM	-	Scanning Electron Microscopy
TGA	-	Thermal Gravimetric Analysis
UTHM	-	Universiti Tun Hussein Onn Malaysia
UTM	-	Universal Testing Machine
UV-vis	-	Ultraviolet visible
WO	-	Waste vegetable oil
XRD	-	X-ray Diffraction Analysis

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CHAPTER 1

INTRODUCTION

1.0 Introduction

Polymers play a significant role in industrial and domestic life, in which the demand for it is always increasing. The raw materials for the production of polymers are derived from petroleum products (oil) and natural gas. The use of petroleum-based monomers in the manufacture of consumer products is expected to decline in coming years because of the continuous rise in the price of oil and the rapid depletion of known oil reserves (Zhang B, 2014; Badri, 2012). Interest has begun with the need to conserve these nonrenewable resources to prepare feedstock for the polymer industry that come from renewable resources, especially from biomass crops (Malaysiana, 2015; Acero, 2014).

Biomass is a renewable resource that can be used as a feedstock to replace fossil oil for the production of bio-fuel and chemicals. It is the most abundant renewable resource on earth, which is the major component of cell walls of plants and can be used as an alternative to fossil fuels (Zhang B, 2014). Biomass is far less expensive than other resources, such as crude oil and natural gas, for energy and chemical production based on energy content (Wei, 2013). With the diminishing supply of crude oil, and environmental concerns, biomass can be converted to liquid and gaseous products that can then be used directly in internal-combustion engines or for organic synthesis. Conversion technologies refer to conversion of unrecyclable solid waste into useful products, such as green fuels and renewable energy. The most familiar and accessible biomass feedstock are oil palm fruit waste, wood, algae,

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